MISSOURI DEPARTMENT OF TRANSPORTATION
MATERIALS
Jefferson City, Missouri

Test Method
MoDOT T46
ANALYSIS OF CEMENT, GROUND GRANULATED BLAST FURNACE SLAG AND FLY ASH

1.0 SCOPE

1.1 This method describes a procedure for the analysis of cement, ground granulated blast furnace slag and fly ash by Atomic Absorption Spectrophotometry for the following elements:

Calcium (CaO)
Silicon (SiO_2)
Aluminum (Al_2O_3)
Iron (Fe_2O_3)
Magnesium (MgO)
Potassium (K_2O)
Sodium (Na_2O)

1.2 Titanium Dioxide(TiO_2) and Phosphorus Pentoxide(P_2O_5) are analyzed using Inductively Couple Plasma Spectrophotometry.

2.0 REAGENTS AND APPARATUS

2.1 Atomic Absorption Spectrophotometer conforming to the specifications set forth in ASTM C 114.

2.2 Graphite crucibles, 7.88 ml capacity, made from purified graphite. These should be pre-ignited in a muffle furnace for 20 minutes at 950C prior to use.

2.3 Clear plastic beakers (Polypropylene), 400 ml capacity.

2.4 Magnetic stirring bars. The length of the bars used should be approximately 1/2 inch less than the inside diameter of the plastic beakers.

2.5 Lithium Metaborate ($LiBO_2$), Reagent Grade, anhydrous.

2.6 Nitric Acid (HNO_3). Sp. Gr. 1.42

2.7 Hydrochloric Acid (HCl), Sp. Gr. 1.19

2.8 Lanthanum Oxide (La_2O_3), 99.99 percent pure, low calcium.

2.9 Lanthanum Solution - 10 percent. Add 200 ml of distilled water to 117.28 g of lanthanum oxide (La_2O_3) in a 2000 ml beaker. While stirring, carefully add 500 ml of hydrochloric acid. When solution is complete, cool to room temperature, transfer to a 1000 ml volumetric flask and dilute to volume with distilled water.

2.10 Standard Samples. A supply of National Bureau of Standards cement samples.

2.11 1000 ppm Calcium Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure calcium compound.

2.12 1000 ppm Silicon Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure silicon compound.

2.13 1000 ppm Aluminum Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure aluminum metal.

2.14 1000 ppm Iron Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure iron compound.

2.15 1000 ppm Magnesium Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure magnesium compound.

2.16 1000 ppm Potassium Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure potassium compound.

2.17 1000 ppm Sodium Stock Solution. This solution can be purchased from a number of sources or it can be prepared in the laboratory from a suitably pure sodium compound.

3.0 PROCEDURE

3.1 Selection of Standards

3.1.1 Select a series of national Bureau of Standards cement samples to bracket the expected concentration of the elements in the cement or fly ash samples. In those cases where standards are unavailable in the correct concentration range, the standard solutions may be adjusted to higher levels of the element of interest by addition of the proper amount of the stock solutions listed in Sections 2.11 through 2.17.

3.2 Preparation of standard solutions and sample solutions.

3.2.1 Weigh 0.80 g of anhydrous $LiBO_2$ into a suitable mixing vessel, then add 0.5000 g of cement standard, cement sample, or fly ash sample. Mix thoroughly and transfer the mixture to a pre-ignited graphite crucible. Place the crucible in the mouth of a muffle furnace and heat at 950C for five minutes (or until the mixture melts). Remove from the furnace and gently swirl to coagulate any particles of fusion mix remaining on the walls of the crucible. Place the crucible within the furnace and heat for 10 minutes. Remove from the furnace and immediately pour the molten melt into a clear polypropylene beaker containing 100 ml of 1:24 HNO_3 and a Teflon-coated magnetic stirring bar. Place the beaker on a magnetic stirring unit and stir for ten to fifteen minutes. Filter through Whatman No. 41 filter paper into a 500 ml volumetric flask. Wash the beaker and filter paper thoroughly with 1:24 HNO_3. Dilute to volume with 1:24 HNO_3, mix well, and transfer to a clean polyethylene bottle. Prepare a fusion-blank solution in the same manner, omitting only the addition of sample.

3.3 Calibrate the instruments using the fusion-blank solution and the standard solutions, then, using the AA Spectrophotometer, determine percent SiO_2, percent Al_2O_3, percent Fe_2O_3, percent K_2O and percent Na_2O on the sample solutions. Determine the percent TiO_2 and the percent P_2O_5 using the ICP Spectrophotometer. Pipette 10 ml of 10% Lanthanum solution, 5 ml of HNO_3, and a 2 ml aliquot of the solutions prepared in Section 3.2.1 into a 100 ml volumetric flask and dilute to volume with distilled water. Determine percent CaO and percent MgO on this dilution.

4.0 CALCULATION AND REPORT

4.1 The method of calculation will vary with the make and model of instrument used. Report the elements determined as follows:

% Silicon Dioxide (SiO_2)
% Aluminum Oxide (Al_2O_3)
% Iron Oxide (Fe_2O_3)
% Calcium Oxide (CaO)
% Magnesium Oxide (MgO)
% Potassium Oxide (K_2O)
% Sodium Oxide (Na_2O)
% Titanium Dioxide (TiO_2)
% Phosphorus Pentoxide (P_2O_5)